

### Remarks

Claims 1, 7, 13-14, 20-21, 23, 25-31, 36-43, 50, 63, 66-67, and 69-70 are amended; claims 1-5, 7-23, 25-31, 36-64, 66-67, and 69-70 are pending in this application.

Applicant would like to thank Examiner John M. Cooney for the courtesy extended during the telephonic interview on December 12, 2006 with Applicants' representatives Monique M. Perdok Shonka and Geoffrey Cooper. Also in attendance during the telephonic interview were Keith D. Strassner and Eric W. Anderson (representatives of the University of Missouri). The Office Action mailed September 14, 2006 and a draft reply were discussed during the interview.

The above account is believed to be a complete and accurate summary of the interviews as required by 37 C.F.R. § 1.133. If the Examiner believes that this summary is inaccurate or incomplete, Applicant respectfully requests that the Examiner point out any deficiencies in his next communication so that Applicant can amend or supplement the interview summary.

All claims reciting a "sol-gel material" are amended to recite a "metal oxide or silicon oxide based sol-gel material," both prior to the step of cross-linking and after the step of cross-linking ("cross-linked metal oxide or silicon oxide based sol-gel material") except in reference to the final product of the claimed method which is now referred to as a "metal oxide or silicon oxide based cross-linked aerogel material." Support for these amendments is found in the instant specification, for example, see page 3, lines 19-20 where the definition of "gel point" discloses that a "network of linked oxide particles spans the container holding the sol" when the gel point is reached; page 3, lines 21-22, wherein "hydrolysis" is stated to be the reaction of metal alkoxides with water to form a metal hydroxide; page 3, lines 10-12, wherein "condensation" is stated to occur when two metal hydroxides combine to give a metal oxide species; page 6, lines 14-18, wherein it is stated how silica is formed from silicon alkoxides, showing hydrolysis /condensation reactions as defined above; page 3, lines 23-26, where in the definition of "sol," it is stated that in the reaction of the sol to yield the gel that the "molecular weight of the oxide species produced continuously increases" during the gelation process; and page 3, line 27 to page 4, line 2, wherein it is discussed that a sol-gel material consists of a solid part and a solvent part, the solid part being formed by the three-dimensional network of linked oxide particles, and in the Examples, where a silicon oxide based material of the invention is described in detail.

Accordingly, this three-dimensional matrix, after cross-linking and drying to provide the final product of the claimed method, is now referred to as a "porous cross-linked metal oxide or silicon oxide based aerogel material." Support for use of this "aerogel" terminology for the final product is found throughout the instant specification.

Independent claims 1 and 25 (and all the remaining pending claims which are dependent thereon; claim 43 is amended herein to depend upon claim 1) recite cross-linking with a cross-linking agent comprising an organic compound and are herein amended to further recite that the cross-linked sol-gel material comprises organic cross-links, that is, cross-links comprising carbon-based moieties. Support for this amendment is found in the instant specification, for example on page 10, lines 30-31, it is stated that a cross-linking agent within the scope of the invention includes compounds that are able to form a chemical bond with at least two reactive groups on a sol-gel material forming a structure of the type  $\text{SiO-A-OSi}$  (page 11, line 4) wherein A is the covalently linked cross-link; therefore the atoms of the carbon-based cross-linking agent are incorporated into the structure. The instant specification further provides examples of organic cross-linking agents such as diisocyanates (organic molecules bearing two  $\text{-N=C=O}$  groups), bis acid chlorides, bis anhydrides, and the like, all containing organic groups that are incorporated into the covalently bound cross-links. Formation of a covalent bond between the metal oxide or silicon oxide (silica) surface and the cross-linking agent necessarily creates organic cross-links, that is, links comprising carbon-based moieties that are covalently incorporated into the sol-gel structure and thus into the final aerogel structure after drying.

The recitation of steps in a defined sequential order in claim 1, first forming an metal oxide or silicon oxide based sol-gel material to provide a preformed metal oxide or silicon oxide based sol-gel material, then cross-linking the preformed sol-gel material, then drying the cross-linked sol-gel material to provide a cross-linked metal oxide or silicon oxide based aerogel, finds support in the instant specification. For instance, in Example 1, tetramethoxysilane (tetramethylorthosilicate) is held in methanol solution with ammonium hydroxide initiator for two days to complete polymerization into silica gel, then the resulting silica gel is transferred into a container and contacted with poly(hexamethylenediisocyanate), after which the cross-linked sol-gel material is solvent-exchanged and then dried to provide the inventive aerogel.

The defined sequential order of steps in claim 25 as amended recites formation of an metal oxide or silicon oxide based sol-gel material, followed by treatment of the preformed sol-gel material with an organic cross-linking agent such that the cross-linked metal oxide or silicon oxide based sol-gel material comprises organic cross-links. A step of drying to provide an inventive aerogel is recited in claim 40 as amended herein. Support for these amendments is likewise found in the instant specification, such as in Example 1.

Claim 39 is amended to compare the resistance to rupture under load of a cross-linked metal oxide or silicon oxide based sol-gel material with that of the material without cross-linking. Support for this is found, for example, in Table 2, where it is shown that as a function of the wt% of the cross-linking reagent (e.g., diisocyanate), resistance to rupture under load increases dramatically. The load at rupture for a non-cross-linked silica-based material is only 0.12 kg, whereas at 4 wt% of the cross-linker, the load at rupture increases to 1.75 kg and at 8 wt% of the cross-linker to 3.95 kg.

Claims 50, 66, 67, 69, and 70 are amended to remove references to pores and areas surrounding pores, so that these claims now refer to "surfaces" without further definition. It is well-known in the art that solid-liquid reactions take place predominantly at the surfaces of the solid, and in this case the solid is a porous three-dimensional network as is stated on page 3, line 27, to page 4, line 2. Further support is found, for example, on page 5, lines 21-23, where it is stated that "the contour surface of silica can be used as a template for the deposition and growth of the interparticle crosslinker."

#### The 35 U.S.C. §112, First and Second Paragraph, Rejections

Claims 25-31, 36-43, 50-64, 66, 67, 69 and 70 were rejected under 35 U.S.C. § 112, first paragraph, as failing to comply with the written description requirement. Applicants respectfully traverse the rejections and, to the extent they are maintained with respect to the claims as amended herein, respectfully request reconsideration and withdrawal of the rejections.

In response to the various grounds of rejection provided by the Examiner in order:

Claim 25 is amended to recite that the crosslinking agent is deposited merely on the surfaces of the preformed sol-gel material, without specification of its proximity to any pores that may exist. Thus, this rejection is deemed to be overcome.

Claim 39 is amended herein to compare the resistance to rupture under load of the cross-linked metal oxide or silicon oxide based sol-gel material with that of the same material without cross-linking. Thus, this rejection is deemed to be overcome.

Claim 50 recites a cross-linked sol-gel material which is filled with a solvent, wherein the solvent is exchanged with a new solvent, followed by drying under non-critical conditions such that the new solvent is removed to provide the dried material. Applicant respectfully submits that support for this claim is found in the instant specification in a manner that does reasonably convey to one skill in the art that the inventor had possession of the invention at the time of filing. Specifically, Example 3 describes a metal oxide or silicon oxide based aerogel material that is prepared without supercritical drying. Prior to the solvent exchange and drying steps, the cross-linked sol-gel material, following gelation of tetramethoxysilane, then cross-linking with poly(hexamethylenediisocyanate), is left filled with acetone resulting from the four eight-hour washes in acetone as is described in Example 1. Then, in Example 3, this material is solvent-exchanged with the new solvent pentane by four twelve-hour washes, followed by drying in an oven at 40° for one hour to yield the inventive aerogel.

Claims 66, 67, 69, and 70 are all amended herein to recite that the surfaces, or the chemical functionality of the surfaces, serve as a template for the cross-linking agent, or as a template for reaction with or accumulation of the cross-linking agent, respectively. It is well-known in the art that liquid-solid reactions take place predominantly at the solid surfaces, where the chemical functional groups involved in the reaction (OH or O<sup>-</sup> groups in the case of silica or metal oxides) are available for reaction with the cross-linking agent, for example, formation of a urethane linkage by reaction of the oxygen groups with isocyanate groups in the cross-linker.

Claims 25-31, 36-43, 69 and 70 were rejected under 35 U.S.C. § 112, first paragraph, because the specification, while being enabling for silica aerogel monoliths prepared from sol-gel materials based on silica, does not reasonably provide enablement for any porous material from any sol-gel material. Applicants respectfully traverse the rejections and, to the extent they are maintained with respect to the claims as amended herein, respectfully request reconsideration and withdrawal of the rejections.

As amended herein, claim 25 and claims 37-42 and 69-70 which depend thereon (claim 36 is cancelled), and dependent claim 43, all recite a "metal oxide or silicon oxide based sol-gel material." The Examiner admits that the specification is enabling for a porous silica-containing solid, i.e., a "silica aerogel monolith" that is prepared from sol-gel materials based on silica. It is well-known in the art that silica is synonymous with silicon oxide; other analogous metal oxides or are also well-known; for example aluminum oxide, magnesium oxide, titanium oxide, and the like, all of which are encompassed by the descriptor "metal oxide or silicon oxide," as is well-known to skilled artisans. Such metal oxides and silicon oxides have similar chemistry, and sol-gel materials formed from analogous precursors to those that are used in the silicon oxide case would be cross-linked by organic cross-linking agents that react with oxygen atoms, in a similar manner as is illustrated in the case of silicon oxides, without requiring undue experimentation. Claim 25 as amended herein recites the formation of an metal oxide or silicon oxide based sol-gel material, as does claim 43, which as amended herein depends upon claim 1. Therefore it is deemed that this rejection is overcome.

Claim 39 was rejected under 35 U.S.C. § 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which Applicant regards as the invention. Applicants respectfully traverse the rejection and, to the extent it is maintained with respect to the claim as amended herein, respectfully request reconsideration and withdrawal of the rejection.

Claim 39, as amended herein, recites that "the cross-linked metal oxide or silicon oxide based sol-gel material is ~~stronger or more robust~~ more resistant to rupture under load than ~~the preformed metal oxide or silicon oxide based~~ sol-gel material." This provides the basis for the comparison of values for rupture under load, as are shown in Table 2 of the Specification, of the cross-linked material in comparison to sol-gel material that has not undergone crosslinking with an organic compound. Therefore, this rejection is deemed to be overcome.

Claims 1-5, 7-23, 25-31, 36-64, 66, 67, 69 and 70 were rejected under 35 U.S.C. § 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which Applicant regards as the invention. Applicants respectfully traverse the

rejections and, to the extent they are maintained with respect to the claims as amended herein, respectfully request reconsideration and withdrawal of the rejections.

Independent claims 1 and 25 (claim 43 is amended herein to depend upon claim 1), and the claims that depend thereon are amended herein to recite a metal oxide or silicon oxide based sol-gel material. Thus, the rejected claims all now recite what materials are included by the meaning of the term, namely those based on metal oxide or silicon oxides as exemplified by silica (silicon dioxide and its hydrates), and including analogous metal oxides, such as aluminum oxide and the like. Therefore, this rejection is deemed to be overcome.

Thus, Applicant respectfully submits that the claims are in conformance with § 112, first and second paragraph. Therefore, withdrawal of the § 112 rejections is respectfully requested.

#### The 35 U.S.C. §102 Rejections

Claims 1, 5, 7-17, 20-22, 25-31, 36, 39-64, 66, 67, 69 and 70 were rejected under 35 U.S.C. § 102(b) as allegedly anticipated by Kawakami et al. (U.S. Patent No. 6,364,953). Applicants respectfully traverse the rejections and, to the extent they are maintained with respect to the claims as amended herein, respectfully request reconsideration and withdrawal of the rejections.

Anticipation requires the disclosure in a single prior art reference of each element of the claim under consideration. *In re Dillon*, 919 F.2d 688, 16 U.S.P.Q.2d 1897, 1908 (Fed. Cir. 1990) (en banc), cert. denied, 500 U.S. 904 (1991). For anticipation, there must be no difference between the claimed invention and the reference disclosure, as viewed by a person of ordinary skill in the art. *Scripps Clinic & Res. Found. v. Genentech, Inc.*, 927 F.2d 1565, 18 USPQ2d 101 (Fed. Cir. 1991).

Claim 1 as amended herein, recites a method to prepare a porous cross-linked metal oxide or silicon oxide based aerogel material, the method comprising: (a) first, forming a metal oxide or silicon oxide based sol-gel material to provide a preformed metal oxide or silicon oxide based sol-gel material; then, (b) contacting the preformed metal oxide or silicon oxide based sol-gel material with a cross-linking agent, the cross-linking agent comprising an organic compound, to provide a cross-linked metal oxide or silicon oxide based sol-gel material comprising organic

cross-links; and then, (c) drying the cross-linked metal oxide or silicon oxide based sol-gel material to form the porous cross-linked metal oxide or silicon oxide based aerogel material.

Claim 1 therefore recites a three-step process comprising an initial step of formation of a sol-gel material, for example formation of silica gel through an ammonia-initiated treatment of tetramethoxysilane; then, subsequent to this reaction and after gelation of the metal oxide or silicon oxide based sol-gel material is complete, there is a separate, second step of cross-linking this preformed sol-gel material with an organic cross-linking agent such that the reaction product, a cross-linked metal oxide or silicon oxide based sol-gel material, comprises organic (carbon-based) cross-links; then, subsequent to the cross-linking step, a step of drying of the cross-linked sol-gel material is carried out to provide the porous cross-linked metal oxide or silicon oxide based aerogel of the invention.

In contrast, Kawakami et al. discuss a method for making an aerogel film that includes the steps of performing a gelation reaction of a silicon oxide to form a wet-gel film on a solid substrate, then a step of performing a step of super-critical or sub-critical drying to provide a silica aerogel film on the substrate. This is a two-step process. Kawakami does not disclose treatment of the wet-gel film with a cross-linking agent comprising an organic compound, whereby an organic cross-link is introduced into the material prior to the step of drying. The gelation promoters of Kawakami (none listed are organic compounds) are not disclosed to cause incorporation of an organic cross-link into the product aerogel. The tetramethylorthosilicate (which Kawakami refers to as a metal alkoxide; for example see column 8, lines 8-9, wherein Kawakami states "[a]lkoxides of tetravalent metals other than silicon also cause gelation"), while it may react with an existing silica structure during the silica gelation process, does not provide for the incorporation of organic cross-links, as it only deposits silicon oxide in its reaction with the silica.

It may be that the Examiner is viewing the incorporation of additional silicon oxide molecules into the growing silica gel framework of Kawakami et al. during the initial gelation reaction as the cross-linking of a preformed gel rather than as additional reactions in the initial formation of the silica gel framework. To clarify what the inventors believe their invention to be, claim 1 as amended herein clarifies that the gelation reaction yielding the oxide based material is already complete and not ongoing when the cross-linking with an organic cross-linker

is carried out as a separate step. The cross-linking reaction results in formation of a cross-linked oxide based sol-gel material comprising organic cross-links, because incorporation of the organic cross-linking agent into the sol-gel structure takes place. Kawakami et al. do not disclose this feature. After cross-linking is complete, the cross-linked sol-gel material is dried to provide the cross-linked oxide based aerogel material. Therefore, Kawakami et al. do not disclose every element of claim 1, and cannot anticipate claim 1.

Claim 25, as amended herein, recites as a composition of matter a cross-linked metal oxide or silicon oxide based sol-gel material, wherein a metal oxide or silicon oxide based sol-gel material is formed to provide a preformed metal oxide or silicon oxide based sol-gel material, then a cross-linking agent comprising an organic compound is deposited on surfaces of the preformed sol-gel material so as to form a cross-linked metal oxide or silicon oxide based sol-gel material comprising organic cross-links. The cross-linking agent comprising an organic compound is deposited on surfaces of the preformed sol-gel material such that the cross-linked metal oxide or silicon oxide based sol-gel material comprises organic, i.e., carbon-containing, cross-links. Kawakami et al. do not disclose this element of claim 25.

Again, Kawakami et al. recite a two-step process for forming an aerogel film--gelation of a silicon-containing precursor, with or without gelation initiators (catalysts) or retarders (inhibitors), followed by solvent removal from the resulting gel. There is no step of contacting the preformed silica gel with an organic cross-linking agent such as to incorporate organic cross-links into the silica gel disclosed in Kawakami et al.. Therefore, Kawakami does not disclose every element of claim 25, and thus cannot anticipate claim 25 and claims dependent thereon.

Thus, Applicants respectfully request withdrawal of the rejections.

Claims 1-5, 8-23, 25-31, 37-64, 66, 67, 69 and 70 were rejected under 35 U.S.C. § 102(b) for anticipation by Biesmans (U.S. Patent No. 5,990,184). Applicants respectfully traverse the rejections and, to the extent they are maintained with respect to the claims as amended herein, respectfully request reconsideration and withdrawal of the rejections.

Biesmans discusses a polyisocyanate based aerogel, that is, an aerogel that contains organic materials derived from reactions of polyisocyanates (i.e., organic molecules containing a plurality of  $-N=C=O$  groups). There is no metal oxide or silicon oxide based sol-gel material



such as silica, or any metal oxide, present in Biesman's aerogel. For example, referring to column 1, lines 36-44, it is stated that "the present invention provides a method for preparing a polyisocyanate based organic aerogel comprising the steps of a) mixing an organic polyisocyanate and an isocyanate trimerisation catalyst in a suitable solvent, b) maintaining said mixture in a quiescent state for a sufficiently long period of time to form a polymeric gel, and c) supercritically drying the obtained gel, wherein a (co)polymer containing at least one isocyanate-reactive group is mixed with the other ingredients in step a)." At column 1, lines 45-55, Biesmans clarifies that suitable classes of (co)polymers are all organic polymers, and thus does not include silica among such substances. It is well known in the art that polyisocyanates are not metal oxide or silicon oxide based sol-gel materials.

Furthermore, in Biesmans, the polyisocyanate, the isocyanate trimerisation catalyst, and the (co)polymer containing at least one isocyanate-reactive group, are all mixed together at once (in step a)), whereas in claim 1 and 25 of the present application, as discussed above, the isocyanate cross-linking agent is deposited on surfaces of the preformed sol-gel material. Biesmans does not disclose the production of a preformed metal oxide or silicon oxide based sol-gel material with subsequent cross-linking by a cross-linking agent that provides organic cross-links in the resulting aerogel. Therefore, the claims of the present application, all of which recite a preformed oxide based sol-gel material that is subsequently contacted with a crosslinking agent comprising an organic compound, are not anticipated by Biesmans. As Biesmans does not disclose every element of the claims, Applicants respectfully request withdrawal of these rejections.

Claims 1-5, 7-23, 25-31, 36-64, 66, 67, 69 and 70 were rejected under 35 U.S.C. § 102(a) for anticipation by Leventis et al. ("Nanoengineering strong silica aerogels"). Applicants respectfully traverse the rejections and request withdrawal of the rejections.

An affidavit under 37 CFR § 1.132 is submitted herewith wherein the inventors of the present application unequivocally assert that the reference discloses subject matter invented only by the applicants herein, Nicholas Leventis and Chariklia Leventis, rather than by the remaining two authors of the cited document, Guohui Zhang and Abdel-Monem M. Rawashdeh, who were students of the inventors and working under their direct supervision. Under MPEP § 716.10,

such an uncontradicted unequivocal statement should be sufficient to establish inventorship.

Therefore, the cited document is not the invention "of another" within the meaning of the term under 35 USC § 102(a), and Applicants respectfully request removal of the document as prior art by the Examiner and withdrawal of the rejections made on this basis.

**Conclusion**

Applicant respectfully submits that the claims are in condition for allowance, and notification to that effect is earnestly requested. The Examiner is invited to telephone Applicant's attorney at (612) 373-6905 to facilitate prosecution of this application.

If necessary, please charge any additional fees or credit overpayment to Deposit Account No. 19-0743.

Respectfully submitted,

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**CERTIFICATE UNDER 37 CFR 1.8:** The undersigned hereby certifies that this correspondence is being filed using the USPTO's electronic filing system EFS-Web, and is addressed to: Mail Stop Amendment, Commissioner of Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on this 13 day of December 2006.

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